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GROWTH OF HgCdTe BY MODIFIED
MOLECULAR BEAM EPITAXY.

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FORWARD

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SUMMARY

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We have designed and constructed an apparatus for the epitaxial growth of HgCdTe compound by a novel approach. This approach is a version of laser induced vacuum evaporation, but the key areas are specially modified to suit the system's need.

The operation involves the use of two lasers. A 100 watt high power YAG laser will be pulsed, beam-splittered and focused onto the component compounds (HgTe and CdTe) causing congruent evaporation. The two evaporants will condense and mix on a slightly heated substrate material to form HgCdTe film of the desired composition. The amorphous film so formed will be crystallized in situ with an Nd⁺²: glass annealing laser which is Q-switched and delivers 20 ns width pulses with peak power exceeding 10⁷ w/cm². The approach is basically a "low temperature" process. There will not be any hot spots in the system to contribute outgassing impurities. The controls will be simple and locate outside the vacuum system.

We have also estimated the power requirement of the evaporating laser. Conservative estimation on the evaporation rate predicts a growth rate greater than 4 $\mu\text{m}/\text{hr}$.

We have also pointed out that there is a difference between the laser annealing in vacuum and in the ambient atmosphere. We have pointed out that in the vacuum, when the peak power is high enough, it can create an instant high pressure over the sample surface. The phenomenon can potentially aid the laser annealing process.



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1.0 INTRODUCTION

1.1 Overall Program Objectives

The main objective of this program is to explore and develop a novel epitaxial technique for the HgCdTe system. Basically, the proposed approach is a form of vacuum deposition similar to the well known MBE. However, due to the high vapor pressure and the rapid interdiffusion of this material conventional MBE is not suitable. We have innovated a number of modifications to accommodate these stringent requirements. Key differences between the two methods are the beam sources and the way to achieve epitaxial annealing. Two alternative beam sources were proposed.

1.2 Overall Program Plans

Because of the pioneering nature of this subject, most parts of the initial efforts involve the system design and construction. This includes the selection and purchasing of capital equipment and the setup of a new laboratory. In addition, we also need an extensive literature search and to carry out some analytic studies. This survey allows us to foresee the potential problems and take them into consideration. Moreover, we can base on this study to decide which one of the two proposed beam sources are more advantageous and more likely to succeed. So far in the course of this program, these tasks are completed or near completion. Our progress is essentially on schedule according to the following phases that were generated before:

- Phase I System Design, Construction and Analytic studies:
 1st and 2nd quarter, completed.
- Phase II Epitaxial Growth and Optimization:
 3rd quarter through the end of the program.
- Phase III Epilayer Characterization:
 4th quarter through the end of the program.



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1.3 Accomplishments in the First Semi-Annual Period

During the first six months, we have completed the following:

- (1) We have selected and purchased the necessary capital equipment. The total cost is over \$65,000.
- (2) We have designed the system. The main vacuum chamber is completely assembled and tested.
- (3) We have made some literature search and analytic studies on this subject. We have also estimated the power requirement for the evaporation laser and the growth rate.

The only unaccomplished goal is the installation of the lasers. This delay is caused by the postponement of the manufacturer's delivery schedule. The laser for evaporation is now set to arrive in early October. Therefore, it is reasonable to expect the first run in late October.



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2.0 TECHNICAL INFORMATION

2.1 General Description

We proposed two means of generating beams of the source materials: a nozzle beam and a laser generated beam. After careful literature survey and design consideration, we decide that the combination of a laser evaporated beam with in situ laser annealing is most viable. During the past six months, this idea has been refined. Now it takes a much more advanced form than the one described in our proposal. In the following sections, we will give a brief description of the system and the approach. Figures 1a and 1b show the pictures of the entire apparatus and the closeup view of the main chamber. Figure 2 shows its schematics diagram. The system consists of three major components.

2.1.1 Main Vacuum Chamber

This unit is already assembled and tested. It is 12" in diameter and 10" tall and has an all metal construction for UHV application. It is backed up by two parallel pumping systems. One is a mercury diffusion pump stack and the other is a Varian diode ion pump. The mercury diffusion pump is equipped with a liquid nitrogen cooled second stage baffle and a thermal electrically cooled first stage baffle. The foreline is also all metal construction and heavily trapped with molecular sieve material to minimize mechanical pump oil backstreaming. Its virtue is the simplicity and the high pumping speed. It requires only 15 minutes to reach a vacuum in the 10^{-7} torr range. It is oil-free and has no foreign contamination except mercury vapor. The diode pump is a 500 L/S model. It can routinely achieve low 10^{-9} torr vacuum. We will grow HgCdTe layer under both vacuum conditions.

In this chamber, we have installed a thickness monitor and a quadrupole mass analyzer for beam monitoring. This analyzer covers a range up to 400 AMU, thus even the heavy evaporated species such as HgTe can be detected.



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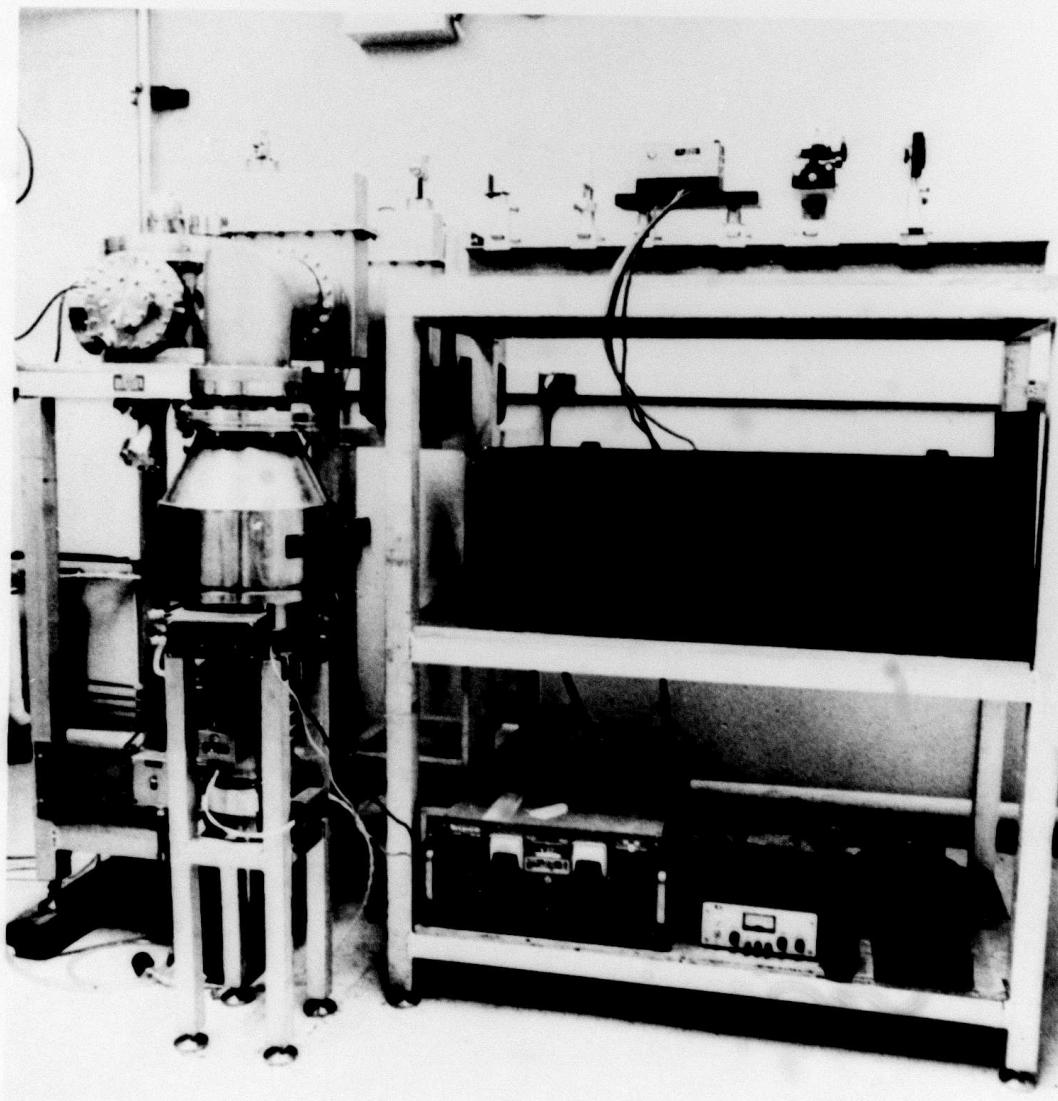


Fig. 1a Photograph of the entire apparatus.



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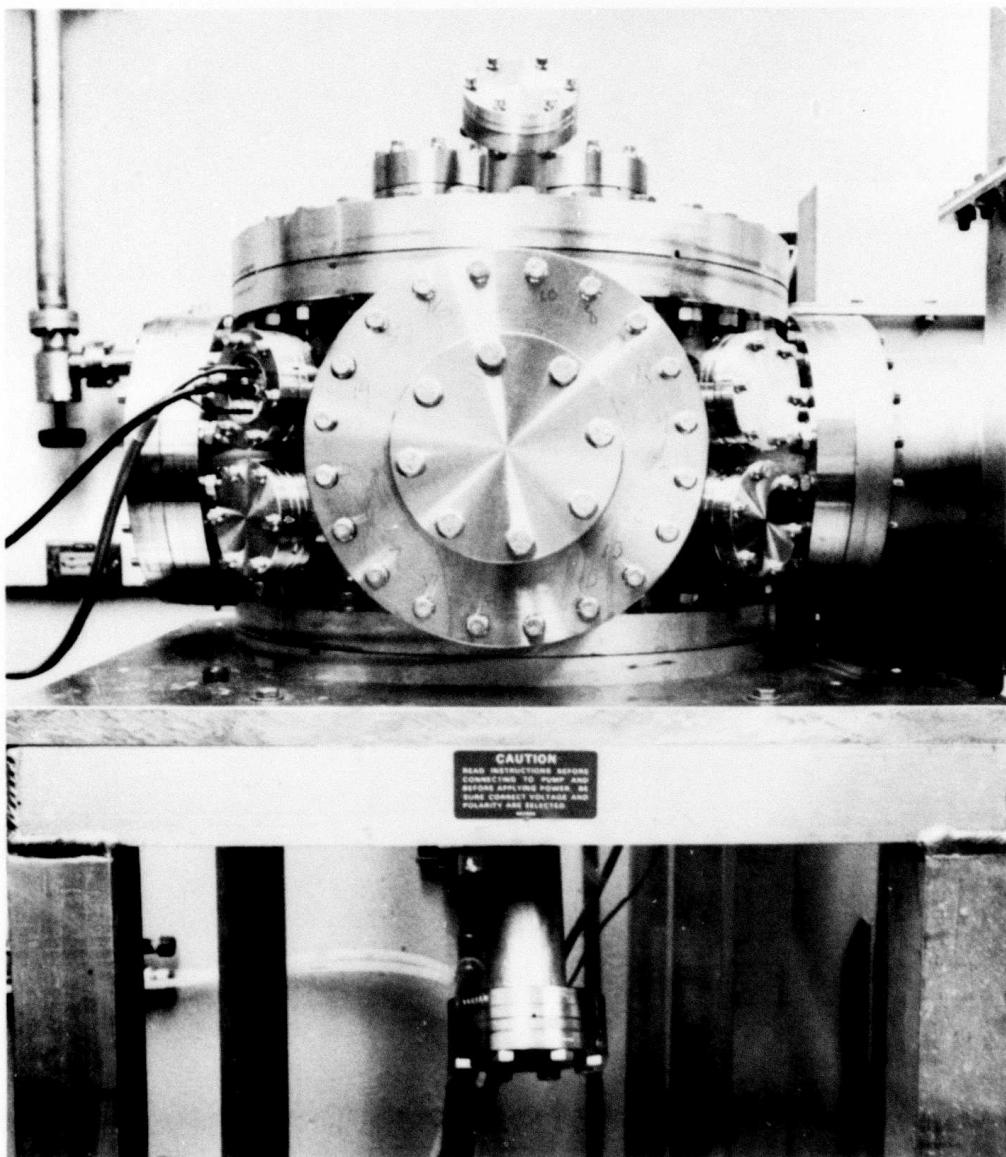


Fig. 1b Close up view of the main chamber.



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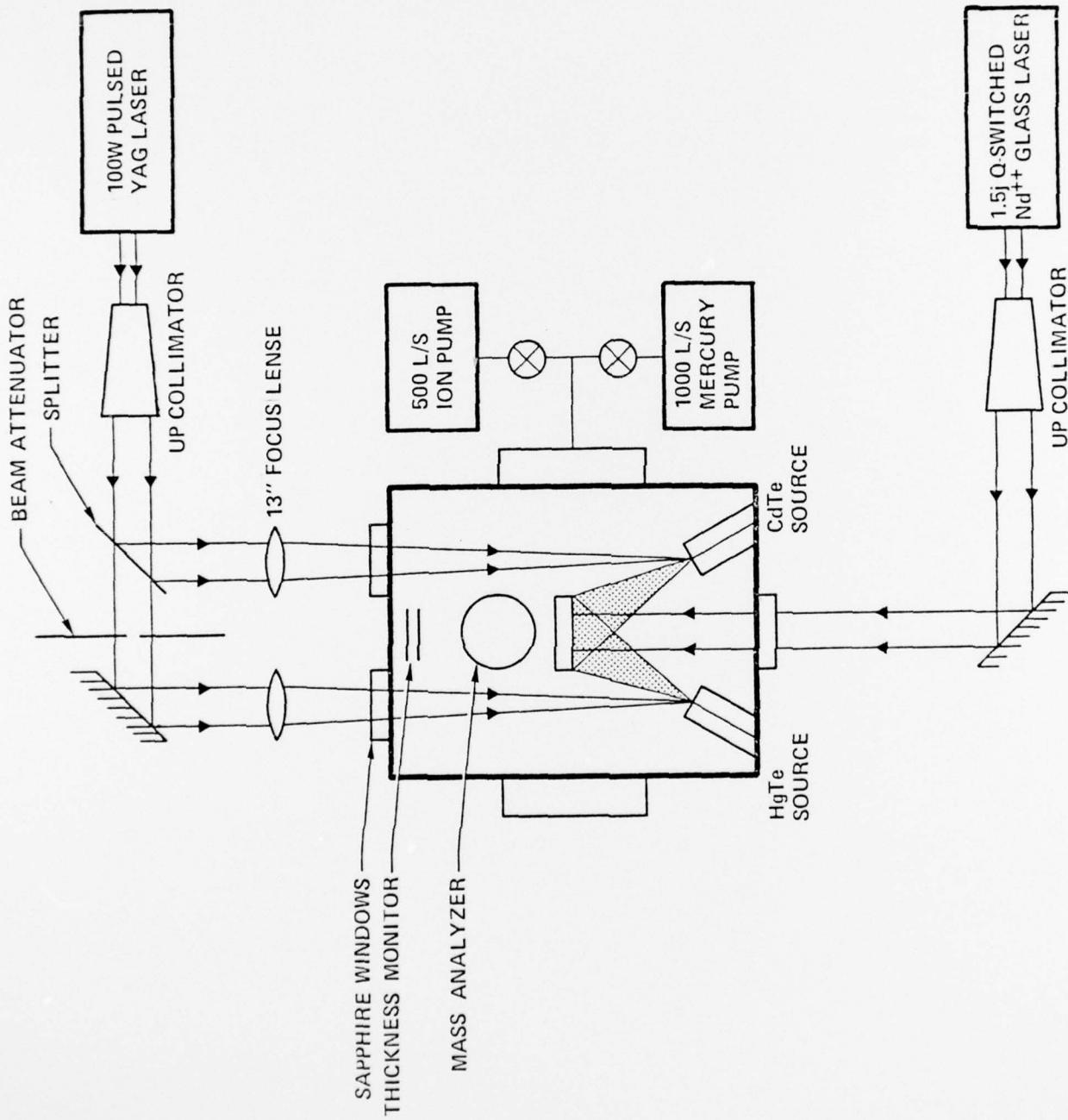


Fig. 2 Schematic diagram of the system.



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Substrate wafers will be loaded through one of the side flanges. Up to four wafers can be mounted on the 10" linear travel during a single loading. This will greatly reduce preparation and pump-down time. The substrate can be heated to a preset temperature. Our plan is to grow the epi-layer at a substrate temperature as high as possible before re-evaporation and out-diffusion occur.

There are two sapphire windows on the top flange and one on the bottom flange. They are for the transmission of evaporation and annealing laser beams, respectively. The transmission of sapphire windows at the 1.06 μ laser wavelength is 90%.

There are three source flanges. Two of them are for HgTe and CdTe compound sources. The third one has a regular MBE effusive furnace. It will be used for future doping experiments.

2.1.2 Evaporation Laser

This unit locates above the top level of the main chamber. It is a Nd-YAG laser (Control Laser Model 514 QT) and can deliver a maximum of 100 W cw power in the multimode operation. It has an acousto-electro Q-switch for pulsed operation. In the pulsed mode, the laser output frequency can vary from 0.5 KHz to a few KHz and the peak power is in few kW range. Together with this unit is a set of custom designed optical components. This includes a 3:1 up collimator, a beam splitter and two 90° steering optics with a set of 13" focal length lenses. Their function will be discussed in a later section.

2.1.3 Annealing Laser

It is placed below the bottom level of the main chamber. It is a Nd-Glass laser (Korad K1) with a Pockel-cell Q-switch. It can deliver extremely narrow (>20 nsec) pulses. The total energy density per pulse is about 1-2 Joule/cm². Thus a 20 ns laser pulse would have a peak power of 10⁷-10⁸ W/cm², a typical value for pulsed laser annealing. The maximum pulse rate is 4 ppm. It will be directed onto the substrate by a 90° beam steering optics.



2.1.4 Experimental Approach

The key differences between our approach and the conventional MBE are the starting source materials, the beam generation and the annealing procedure. We use molecular compound source (HgTe and CdTe) instead of elemental sources (Hg, Cd and Te₂). The beams are generated by pulsed laser evaporation instead of thermal evaporation. The epitaxial annealing is achieved by in situ pulsed laser radiation instead of thermally heating the substrate.

A pulsed laser beam ($\lambda = 1.06 \mu\text{m}$) emerges from the Nd-YAG laser. The beam is expanded by a 3:1 up collimator. Part of the beam is then directed downward by a splitter and focused onto the CdTe source by a 13" focal length lens. The transmitted beam will be attenuated and bent through 90° and focused onto the HgTe source. The divergence of the laser output is approximately 2.5 mrad, therefore the focused spots are about 0.15 mm in diameter. The irradiated material absorbs the laser power. Because the laser pulses are short and their peak power high, one would expect congruent evaporation. One question that immediately rises is that since CdTe is transparent at 1.06 μm , then how can this work efficiently? However, when the peak power density of the focused laser spot reaches very high levels ($\sim 10^8 \text{ W/cm}^2$), there is no difference in initially opaque and transparent substance. All materials, including transparent dielectrics pass quickly into a highly absorbing state due to optical avalanche breakdown and subsequent heating to light absorption. In the worst case, if CdTe does not evaporate at all, we will then replace it with a short cut-off wavelength HgCdTe bulk material for source. Two source beams will co-deposit onto the substrate to form a film of Hg_xCd_{1-x}Te alloy. Substrate temperature will be kept just below the occurrence of re-evaporation and out-diffusion. Film crystallinity will be restored by Q-switched laser pulses at regular time intervals.



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This approach has three obvious advantages:

- (1) During the process, the only hot spots are the areas radiated by the Nd-YAG laser beam. These are very small ($\sim 0.2 \text{ mm}^2$), thus contribute very little outgassing. Therefore, the use of a pulsed laser as a source of power external to the vacuum system minimize the contamination due to the proximity of hot filaments, crucibles, etc.
- (2) All growth parameter can be easily controlled outside the vacuum system.
- (3) The alloy composition χ is determined by the relative intensity of the two laser beams, or more specifically, by the beam attenuator. Since two beams originate from the same laser unit, any small power fluctuation would only have negligible secondary effect.
- (4) It is a low temperature growth technique.

2.2 Literature Search and Analytic Studies

We have carried out an extensive literature search and analytic studies in the area of laser evaporation. Pioneering work in this area began almost a decade ago. The number of publications are quite limited and they all belong to the small scale research oriented type. A wide range of materials have been used. These include metals, alloys, semiconductor (III-V and II-VI compounds) and dielectric materials. Some most important experiments are summarized in Table I.

These investigations can be categorized into two areas. One is the evaporation with a cw laser and the other with a pulsed laser. Their results are very different. The cw laser operation resembles the use of an E-beam. Here, the source material is heated slowly and continuously by the laser beam until a temperature high enough for evaporation. Compound material evaporated



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REF	TYPE OF LASER	SOURCE MATERIAL	SUBSTRATE	RESULTS
1	CO ₂ , CW, 60W	Dielectric Materials	Quartz	Evaporated films showed good optical properties
2	CO ₂ , CW, 50W Ruby, pulsed, 10 ⁸ W/cm ² peak power	GaP, GaAs, GaSb III-V Compounds (GaP, InAs....)	NaCl	Amorphous, uniform, free of pinholes Polycrystalline, uniform and concurrent
3	Rd-Glass, pulsed, 10J	Metal Alloy	-	Stoichiometric film
4	CO ₂ , pulsed	Sb ₂ O ₃	GaAs	Stoichiometric film
5	CO ₂ , pulsed	Dielectric Materials CdS, GaAs	Quartz	Good optical property

Fig. 3 Literature on laser evaporation.



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this way usually result in a non-stoichiometric and amorphous film. The pulsed laser operation is reminiscent of the standard flash evaporation technique. Except this approach offers better control, smaller amount of evaporated material per pulse, faster heating time and higher surface temperature. This method produces stoichiometric and polycrystalline film and it will be the ideal candidate for the system.

2.2.1 Estimate the Power Requirement and Deposition Rate

The evaporation mechanism can be summarized as the following. When laser radiation is absorbed at the surface of an opaque condensed matter, heat is developed in a thin layer under the surface. As a result, the surface temperature increases. Simultaneous heat conduction takes place into the interior of the material, thus increasing the thickness of the heated layer. Since the velocity of heat transport due to heat conduction decreases with time, thus the heat conduction does not permit an adequate outlet of heat into the interior. Hence the temperature at and near the surface will increase up to time when evaporation commences. From then on the surface temperature is governed by the evaporation mechanism only. Heat conduction ceases to play a significant role in the heat balance.

The minimal laser power density Q_e for evaporation may be estimated in the following way. The thickness of the heated layer at time t after the beginning of the laser pulse is of order $(At)^{1/2}$ where A is the thermal diffusivity coefficient. The thermal energy made available until then is $Q_0 t$ where Q_0 is the laser power density. Thus the material of heated layer has gained an average thermal energy density of $Q_0 t / \rho_0 (At)^{1/2}$, where ρ_0 is the density. For evaporation to take place, this thermal energy density should exceed the sublimation energy (per unit weight) U and the time t should be less than the duration τ of the laser pulse. This leads to the condition:

$$Q_0 > Q_e = \rho_0 U A^{1/2} \tau^{-1/2} \quad (1)$$



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For CdTe, $\rho_0 = 5.7 \text{ g/cm}^3$, $U = 2.4 \times 10^{10} \text{ erg/gram}$, $A = 0.02 \text{ cm}^2/\text{sec}$, if the pulse duration is 100 nsec or $\tau = 10^{-7} \text{ sec}$ then

$$Q_e = \rho_0 U A^{1/2} \tau^{-1/2} = 5.2 \times 10^6 \text{ watts/cm}^2 \quad (2)$$

The focused spot has an area of $3 \times 10^{-4} \text{ cm}^2$, therefore, the minimal average power per pulse for evaporation is

$$Q_0' = 5.2 \times 10^6 \times 3 \times 10^{-4} = 1.5 \text{ kW} \quad (3)$$

This value is well within the power limit of our Nd-YAG laser. HgTe should be easier to be evaporated than CdTe.

We can also make a very crude estimation on the growth rate based on this information. The area is about $5 \times 10^{-4} \text{ cm}^2$ and the depth is $(At)^{1/2}$. Therefore, the total volume of material evaporated per pulse is

$$V = 5 \times 10^{-4} \times (0.02 \times 10^{-7})^{1/2} \\ = 2 \times 10^{-8} \text{ cm}^3$$

Or 3×10^{14} molecules/pulse. If the laser is to be operated at 1 KHz then the total evaporated flux will be 3×10^{17} molecules/sec. If only 1% of the evaporated material is to be intercepted by the substrate (1 cm^2) then the growth rate will be about 3×10^{15} molecules/sec-cm 2 or $4 \mu\text{m/hr}$. This is a very practical value.

2.2.2 Anticipated Problems and Solutions

We can foresee two basic problems with laser evaporation which can be avoided by proper design.

First, one has to avoid prolong exposure of a same region to laser pulses, otherwise this area will reach very high temperature slowly and cause



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noncongruent evaporation. This problem can be easily solved by moving the laser beam and rotating the source material simultaneously. Thus the laser spot can actually scan over the entire source area.

Second problem is the fogging of the transmission window due to the condensation of evaporated materials. We take the following precaution steps to avoid this

- (1) Make the distance between the source material and the window much longer than the distance between the source and the substrate.
- (2) Collimate the region between the source and the window.
- (3) Protect the window by placing a piece of heated quartz plate in front of the window at 45° to the beam. This heated quartz plate will deflect the evaporated material moving toward the window.

2.2.3 Some Remarks on the In Situ Laser Annealing

Up to date, all the work in laser annealing was carried out in the ambient atmosphere. However, we feel that by carrying out the in situ annealing in vacuum, there is a special phenomenon which can potentially benefit the annealing process. When a beam pulse with an energy flux of about 10^7 W/cm² interacts with the surface, it will produce some vapor which escapes in the form of a rapidly rising plume. This plume exerts a recoil pressure on the vaporizing condensed phase. Depending on the condition, the pressure can vary from a few atm to over 1000 atm! If this does happen in our case, it can have similar effect as the use of overpressure during a thermal annealing. During the annealing pulse, the HgCdTe surface will experience a localized "instant" high pressure which can reduce the out-diffusion problem. We will study this and try to utilize it.



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3.0 FUTURE PLANS

We will start to "fire up" the apparatus next month. During the 3rd quarter, we will debug the system and establish a routine operating procedure. Starting from the 4th quarter, we will characterize the epilayers and optimize the growth condition. This effort will be continued until the end of this program. During this period of time, scientifically significant studies such as the mechanism of evaporation, the effect on the beam energy etc. will also be examined.



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